भारतीय मानक
Indian Standard

IS 19004 : 2023

सोडियम हेक्सा मेटा फॉस्फेट, खाद्य ग्रेड – विशिष्टि

Sodium Hexa Meta Phosphate, Food Grade – Specification

ICS 67.220.20

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भारतीय मानक ब्यूरो
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January 2023 **Price Group 6**

Food Additives Sectional Committee, FAD 08

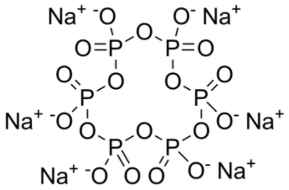
FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

Food additives are added to improve the appearance, flavour, texture or storage properties of the processed foods. As impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. Sodium hexa meta phosphate is used as an emulsifier, sequestrant, texturizer, water softening agent, and moisture binding agent in Pharmaceuticals & Personal Care Products, Poultry Products, Sea foods, Milk and Dairy Products, Fruit and Vegetables processing industries.

This Indian Standard is based on Joint FAO/WHO expert Committee on Food Additives (JECFA) standard. In the preparation of this standard, due consideration has been given to the Legal Metrology (Packaged Commodities) Rules, 2011 & *Food Safety and Standards Act*, 2006 and Rules framed thereunder. The standard is however subject to restrictions imposed under these rules wherever applicable.

Synonym - Sodium hexametaphosphate, sodium tetra polyphosphate, Graham's salt
Chemical name - Sodium Hexa Meta Phosphate,
C.A.S. No. - 68915-31-1, 10124-56-8, 10361-03-2
Chemical formula - $(NaPO_3)_6$
Molecular weight - 611.77
Structural formula –



The composition of the Committee responsible for the formulation of this standard is given in Annex J.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rounding of numerical values (*second revision*)’. The number of significant places retained in he rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SODIUM HEXA META PHOSPHATE, FOOD GRADE — SPECIFICATION

1 SCOPE

This standard prescribes requirements, methods of sampling and test for sodium hexa meta phosphate, food grade.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
IS 1070 : 1992	Reagent grade water (*third revision*)
IS 1699 : 1995	Methods of sampling and test for food colours (*second revision*)
IS 2491 : 2013	Food hygiene – General principles – Code of practice (*third revision*)
IS 2711 : 1979	Direct reading *p*H meters

3 REQUIREMENTS

3.1 Description — Sodium hexa meta phosphate shall be a colourless, glassy, hygroscopic powder.

3.2 The material shall also conform to the requirements given in Table 1.

3.3 The material shall be processed, packed, stored and distributed under hygienic conditions (*see* IS 2491).

4 PACKING, STORAGE AND MARKING

4.1 Packing

The material shall be securely packed in well-filled containers with minimum access to air and moisture because the material is highly hygroscopic. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

4.2 Storage

The material shall be stored in a cool and dry place so as to avoid excessive exposure to heat and moisture.

4.3 Marking

Each container shall be legibly and indelibly marked with the following information:

a) Name of the material including the words ' Food Grade';

b) Name and address of the manufacturer;

c) Batch or Code number;

d) Net quantity when packed;

e) Instruction for storage;

f) Best before date (Month & Year to be given by the manufacturer); and

g) Any other requirements as specified under the Legal Metrology (Packaged Commodities) Rules, 2011 and Food Safety and Standards (Packaging and Labelling) Regulation, 2011 and the Rules framed thereunder.

4.3.1 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provision of *Bureau of Indian Standards Act*, 2016 and Rules and Regulation framed there under and the product(s) may be marked with the Standard Mark.

5 SAMPLING

5.1 Representatives samples of the material shall be drawn according to the method prescribed in **4** of IS 1699.

6 QUALITY OF REAGENTS

6.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE – ‘Pure chemicals’ shall mean chemicals that do not contain impurities, which affect the experimental results.

Table 1 Requirements for Sodium Hexa Meta Phosphate
(*Clause* 3.2)

Sl. No. (1)	Characteristics (2)	Requirement (3)	Method of Test, Ref to (4)
i)	Purity as P_2O_5, percent by mass, on dry basis	67.0 – 68.0	Annex A
ii)	Solubility in water	To pass the test	Annex B
iii)	*p*H	5.8 – 6.6	Annex C
iv)	Orthophosphate	To pass the test	Annex D
v)	Sodium	1 in 20 solution passes test	Annex E
vi)	Loss on ignition, percent by mass, *Max*	1.0	Annex F
vii)	Matter insoluble in water, percent by mass, *Max*	0.1	Annex G
viii)	Fluoride, parts per million, *Max*	10.0	Annex H
ix)	Arsenic (as As), parts per million, *Max*	3.0	**15** of IS 1699
x)	Lead (as Pb), parts per million, *Max*	4.0	**15** of IS 1699

ANNEX A
[*Table* 1, *Sl No.* i)]

METHOD OF ASSAY OF SODIUM HEXA META PHOSPHATE

A-1 APPARATUS

A- 1.1 Standard Volumetric Flask

A- 1.2 Pipette

A- 1.3 Beaker

A- 1.4 Gooch Crucible

A- 1.5 Whatman No. 40 Filter Paper/Circles

A-2 PREPARATION OF SAMPLE

Dry about 10 g of sodium hexa meta phosphate at 110 °C overnight. Keep in a desiccator and bring to room temperature. Take 1 g (accurately weighed) in a 100 ml beaker.

A-3 REAGENTS

A-3.1 Ammonium Molybdate (10 percent w/v)

Dissolve 10 g of ammonium molybdate $[(NH_4)_6Mo_7O_{24}.4H_2O]$ in 100 ml distilled water.

A-3.2 Dilute Nitric Acid (≈ 2 N)

Mix 190 ml of 50 percent nitric acid with distilled water to produce 1 000 ml.

A-3.3 Nitric Acid (2 percent)

Dilute 14 ml of conc. Nitric acid (70 percent) to 710 ml with distilled water.

A-3.4 Potassium Nitrate (1 percent)

Dissolve 1 g potassium nitrate in 100 ml of distilled water.

A-3.5 Phenolphthalein

Dissolve 0.2 g of Phenolphthalein ($C_{20}H_{14}O_4$) in 60 ml of 90 percent ethanol and add sufficient distilled water to produce 100 ml.

A-3.6 1 N Sodium Hydroxide

Dissolve 4g Sodium hydroxide in 100 ml distilled water. Prepare fresh daily for use.

A-3.7 1 N Sulphuric Acid

Dilute 3 ml of concentrated sulphuric acid to 100 ml in distilled water.

A-4 PROCEDURE

Dry about 10 g of sodium hexa meta phosphate at 110 °C overnight. Keep in a desiccator and bring to room temperature. Take 1 g (accurately weighed) in a 100 ml beaker. Dissolve in 25 ml of distilled water. Transfer to a 500 ml standard volumetric flask. Make up the volume to 500 ml with distilled water (1 g sodium hexa meta phosphate/500 ml). Pipette out exactly 25 ml (50 mg sodium hexa meta phosphate) into a 500 ml beaker. Add 5 ml of conc. nitric acid and stir well. Add 220 ml of distilled water kept at 60 °C . Stir well. Wait for 10 min. Add 10 g of ammonium nitrate (purified). At 50 °C , add 100 ml of 10 percent ammonium molybdate (freshly prepared) and stir well with a glass rod for 10 min. Repeat stirring after 1 minute gap. Keep for 30 min. Filter the precipitate using Whatman No. 40 circles and wash with 10 ml of dilute nitric acid (≈ 2 N), followed by potassium nitrate solution (1 percent) until the filtrate is no longer acidic to litmus (Blue litmus remains blue). Dissolve the precipitate from the filter paper in 60 ml of 1 N sodium hydroxide, add phenolphthalein, and titrate the excess sodium hydroxide with 1 N sulphuric acid. Calculate the volume of 1 N sodium hydroxide consumed by the precipitate. Each ml of 1 N sodium hydroxide consumed is equivalent to 0.003 086 g of P_2O_5.

A-5 CALCULATION

Sodium Hexa Meta Phosphate (percent P_2O_5)

Phosphates as P_2O_5, percent by mass on dry basis=

$$\left(60 - \frac{V_1 \times N_1}{N_2}\right) \times \frac{0.06172}{W} \times 100$$

where

V_1 = volume of H_2SO_4 (titre value),
N_1 = normality of H_2SO_4,
N_2 = normality of NaOH,
W = mass in g of the material taken for the test.

Alternatively calculate the P_2O_5 content as follows; P_2O_5 content of sodium hexa meta phosphate = (‘x’ × 6.2) %

where

‘x’ is the volume of 1N NaOH (‘x’= 60 ml – unused volume of 1N NaOH) for example, if ‘x’ is equal to 11 ml, P_2O_5 content of SHMP = 11 × 6.2 = 68.2 %.

ANNEX B

[*Table* 1, *Sl No.* ii)]

TEST FOR SOLUBILITY

B-1 APPARATUS

B-1.1 Test Tube

B-1.2 Graduated Pipette with Least Count of 0.1 ml

B-2 REAGENT

B-2.1 Distilled Water

B-3 PROCEDURE

Transfer 1g of the sample into a test tube containing 1.7 ml of the distilled water, shake for not less than 30 s and no more than 5 min. The sample shall be freely soluble in the solvent.

ANNEX C

[*Table* 1, *Sl No.* iii)]

DETERMINATION OF *p*H

C-1 APPARATUS

C-1.1 *p*H Meter

Use a standard laboratory *p*H meter (*see* IS 2711).

C-2 REAGENT

C-2.1 Distilled Water

C-3 PROCEDURE

Dissolve 20 g of the sample in 100 ml water. Measure the *p*H of the solution using a standard *p*H meter. Repeat the test and record the mean *p*H value.

ANNEX D

[*Table* 1, *Sl No.* iv)]

TEST FOR ORTHO PHOSPHATE

D-1 REAGENTS

D-1.1 Dilute Nitric Acid Test Solution

A solution containing about 10 percent *w/v* of HNO_3. Prepared by diluting 105 ml of nitric acid (70 percent) with water to make 1 000 ml.

D-1.2 Silver Nitrate Test Solution

A 4.2 percent *w/v* solution of silver nitrate ($AgNO_3$) in water (approximately 0.25 N).

D-1.3 Sodium Hydroxide Test Solution

Dissolve 4.3 g of sodium hydroxide in water to make 100 ml (approximately 1 N).

D-2 PROCEDURE

Dissolve 0.1 g of the sample in 5 ml of hot dilute nitric acid test solution. Warm on a steam bath for 10 min, and cool. Neutralize to litmus with sodium hydroxide test solution, and add silver nitrate test solution. A yellow precipitate is formed which is soluble in dilute nitric acid test solution.

ANNEX E

[*Table* 1, *Sl No.* v)]

TEST FOR SODIUM

E-1 REAGENTS

E-1.1 Cobalt-Uranyl Acetate Test Solution

Dissolve, with warming, 40 g of uranyl acetate [$UO_2(C_2H_3O_2)_2.2H_2O$] in a mixture of 30 g glacial acetic acid and sufficient water to make 500 ml. Similarly, prepare a solution containing 200 g of cobaltous acetate [$Co(C_2H_3O_2)_2.4H_2O$] in a mixture of 30 g of glacial acetic acid and sufficient water to make 500 ml. Mix the two solutions while still warm, and cool to 20 °C. Maintain the temperature at 20 °C for about 2 h to separate the excess salts from solution, and then filter through a dry filter.

E-2 PROCEDURE

Sodium compounds, after conversion to chloride or nitrate, yield with cobalt-uranyl acetate test solution a golden-yellow precipitate, which forms after several min agitation. Sodium compounds impart an intense yellow colour to a non-luminous flame.

ANNEX F

[*Table* 1, *Sl No.* vi)]

LOSS ON IGNITION

F-1 PROCEDURE

F-1.1 Take 1 g to 2 g of the substance, previously well mixed and accurately weighed. Reduce the sample to a fine powder when it occurs as crystals. Tare a glass-stoppered, shallow weighing bottle that has been dried for 30 min under the same conditions as will be employed in the determination.

F-1.2 Transfer the sample into the bottle, replace the cover, and weigh the bottle and the sample. Distribute the sample as evenly as practicable to a depth of about 5 mm, and not over 10 mm in the case of bulky materials. Place the bottle with its contents in the drying chamber, removing the stopper and leaving it also in the chamber, and dry the sample at the 135 °C ± 2 °C, for 3 h. Upon opening the chamber, close the bottle promptly and allow it to come to room temperature in a desiccator before weighing. If the substance melts at a lower temperature than that specified for the determination, prepare the sample as described above, then place it in a vacuum desiccator containing sulphuric acid. Evacuate the desiccator to 130 Pa (1 mm of mercury), maintain this vacuum for 24 h, and then weigh the dried sample.

NOTE — However, unless otherwise directed, ignite the sample at a temperature of 450 °C – 550 °C and use a platinum, quartz or porcelain dish instead of the weighing bottle.

ANNEX G
[*Table* 1, *Sl No.* vii)]

DETERMINATION OF WATER INSOLUBLE MATTER

G -1 PROCEDURE

Dissolve about 10 g of the sample, accurately weighed, in 100 ml of hot water, and filter through a tared filtering crucible. Wash the insoluble residue with hot water, dry at 105 °C for 2 h, and weigh.

G-2 CALCULATION

Water insoluble matter, percent by mass

$$= \frac{M_2 - M_1}{M} \times 100$$

where

M_1 = weight of empty Gooch crucible,

M_2 = weight in g, of Gooch crucible with residue,

M = weight in g, of material taken for test,

ANNEX H
[*Table* 1, *Sl No.* viii)]

DETERMINATION OF FLUORIDE

H-1 METHOD I — THORIUM NITRATE COLORIMETRIC METHOD

H-1.1 Treatment of Apparatus:

To minimize the distillation blank resulting from fluoride leached from the glassware, treat the glassware with hot 10 percent sodium hydroxide solution, followed by flushing with tap water and rinsing with distilled water. At least once daily, treat in addition by boiling down 15 ml to 20 ml of dilute sulphuric acid (1 in 2) until the still is filled with fumes; cool, pour off the acid, treat again with 10 percent sodium hydroxide solution, and rinse thoroughly.

H-1.2 Reagents

H-1.2.1 *Perchloric Acid*

H-1.2.2 *Silver Nitrate Solution (1 In 2)*

H-1.2.3 *Sodium Alizarin Sulphonate (1 In 1 000)*

H-1.2.4 *Hydroxylamine Hydrochloride Solution (1 In 4 000)*

H-1.2.5 *1 N or 0.05 N Sodium Hydroxide Solution*

H-1.2.6 *0.1 N Hydrochloric Acid*

H-1.2.7 *Thorium Nitrate Solution (1 in 4 000)*

H-1.2.8 *Sodium Fluoride Test Solution* (*10 µg F per ml*)

Transfer 2.210 g of sodium fluoride, previously dried at 200 °C for 4 h and accurately weighed, into a 400 ml plastic beaker, add 200 ml of water, and stir until dissolved. Quantitatively transfer this solution into a 1 000 ml volumetric flask with the aid of water, dilute to volume with water, and mix. Store this stock solution in a plastic bottle. On the day of use, transfer 2.5 ml of the stock solution into a 1 000 ml volumetric flask, dilute to volume with water, and mix.

H-1.3 Procedure

H- 1.3.1 Place a 5.0 g sample and 30 ml of water in a 125 ml distillation flask having a side arm and trap. The flask is connected with a condenser and carries a thermometer and a capillary tube, both of which must extend into the liquid. Slowly add, with continuous stirring, 10 ml of perchloric acid, and then add 2 drops or 3 drops of silver nitrate solution (1 in 2) and a few glass beads. Connect a small dropping funnel or a steam generator to the capillary tube. Support the flask on an asbestos mat with a hole that exposes about one - third of the flask to the flame. Distil until the temperature reaches 135 °C. Add water from the funnel or introduce steam through the capillary, maintaining the temperature between 135 °C and 140 °C at all times. Continue the distillation until 100 ml of distillate has been collected. After the 100 ml portion (Distillate A) is collected, collect an additional 50 ml portion of distillate (Distillate B) to ensure that all of the fluorine has been volatilized. Place 50 ml of Distillate A in a 50 ml Nessler tube.

H- 1.3.2 In another similar Nessler tube place 50 ml of water distilled through the apparatus as a control. Add to each tube 0.1 ml of a filtered solution of sodium alizarin sulphonate (1 in 1 000) and 1 ml of freshly prepared hydroxylamine hydrochloride solution (1 in 4 000), and mix well. Add, drop wise and with stirring, either 1 N or 0.05 N sodium hydroxide, depending upon the expected volume of volatile acid distilling over, to the tube containing the distillate until its colour just matches that of the control, which is faintly pink. Then add to each tube 1.0 ml of 0.1 N hydrochloric acid, and mix well.

From a burette, graduated in 0.05 ml, add slowly to the tube containing the distillate enough thorium nitrate solution (1 in 4 000) so that, after mixing, the colour of the liquid just changes to a faint pink. Note the volume of the solution added, then add exactly the same volume to the control, and mix. Now add to the control solution, sodium fluoride test solution (10 µg F per ml) from a burette to make the colours of the two tubes match after dilution to the same volume.

H- 1.3.3 Mix well, and allow all air bubbles to escape before making the final colour comparison. Check the endpoint by adding 1 or 2 drops of sodium fluoride test solution to the control. A distinct change in colour should take place. Note the volume of sodium fluoride test solution added. Dilute Distillate B to 100 ml, and mix well. Place 50 ml of this solution in a 50 ml Nessler tube, and follow the procedure used above for Distillate A. The total volume of sodium fluoride test solution required for the solutions from both Distillate A and Distillate B should not exceed 2.5 ml.

NOTE – When applying this test to organic compounds, the temperature at which the distillation is conducted must be rigidly controlled at all times to the recommended range of 135 °C to 140 °C to avoid the possibility of explosion.

H-2 METHOD II — ION-SELECTIVE ELECTRODE METHOD

H-2.1 Reagents

H-2.1.1 *Sodium Fluoride Solution (5 µg F per ml)*

Transfer 2.210 g of sodium fluoride, previously dried at 200 °C for 4 h and accurately weighed, into a 400 ml plastic beaker, add 200 ml of water, and stir until dissolved. Quantitatively transfer this solution into a 1 000 ml volumetric flask with the aid of water, dilute to volume with water, and mix. Store this stock solution in a plastic bottle. On the day of use, transfer 5.0 ml of the stock solution into a 1 000 ml volumetric flask, dilute to volume with water, and mix.

H-2.2 Standard Curve

Transfer into separate 250 ml plastic beakers 1.0, 2.0, 3.0, 5.0, 10.0, and 15.0 ml of the sodium fluoride solution, add 50 ml of water, 5 ml of 1 N hydrochloric acid, 10 ml of 1 M sodium citrate, and 10 ml of 0.2 M disodium EDTA to each beaker, and mix. Transfer each solution into separate 100 ml volumetric flasks, dilute to volume with water, and mix. Transfer a 50 ml portion of each solution into separate 125 ml plastic beakers, and measure the potential of each solution with a suitable ion-selective electrode, using a suitable reference electrode. Plot the standard curve on two-cycle semi-logarithmic paper with µg F per 100 ml solution on the logarithmic scale.

H-2.3 Procedure

Transfer 1 g of the sample into a 150 ml glass beaker, add 10 ml of water, and while stirring continuously, add 20 ml of 1 N hydrochloric acid slowly to dissolve the sample. Boil rapidly for 1 min, then transfer into a 250 ml plastic beaker, and cool rapidly in ice water. Add 15 ml of 1 M sodium citrate and 10 ml of 0.2 M disodium EDTA, and mix.

Adjust the *p*H to 5.5 with 1 N hydrochloric acid or 1 M sodium hydroxide, if necessary, then transfer into a 100 ml volumetric flask, dilute to volume with water, and mix. Transfer a 50 ml portion of this solution into a 125 ml plastic beaker and measure the potential of the solution with the apparatus described under standard curve. Determine the fluoride content, in µg, of the sample from the standard curve.

ANNEX J

(*Foreword*)

COMMITTEE COMPOSITION

Food Additives Sectional Committee, FAD 08

Organization	*Representative(s)*
CSIR-Indian Institute of Toxicology Research, Lucknow	Dr Yogeshwar Shukla (***Chairperson***)
All India Food Processors Association, (India)	Ms Shreya Pandey Shri Krishna Kumar Joshi (*Alternate*)
Association of Food Scientists and Technologists India, Mumbai	Dr Vikas Singh Chauhan Dr Nandini P. Shetty (*Alternate*)
Bose Institute, Kolkata	Professor Gaourishnkar
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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the website-www.bis.gov.in or www.standardsbis.in.

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